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Indian Standard

METHODS FOR SAMPLING OF COAL AND COKE

PART II SAMPLING OF COKE

(Revised)

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

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March 1966

Indian Standard
METHODS FOR SAMPLING OF COAL AND COKE
PART II SAMPLING OF COKE
(Revised)

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METHODS FOR SAMPLING OF COAL AND COKE

PART II SAMPLING OF COKE

(Revised)

0. FOREWORD

0.1 This Indian Standard (Part II) (Revised) was adopted by the Indian Standards Institution on 20 October 1965, after the draft finalized by the Solid Mineral Fuels Sectional Committee had been approved by the Chemical Division Council.

0.2 This standard was originally issued in 1953 with a view to prescribing the procedures for obtaining representative samples of coal and coke for test purposes. Since then extensive investigation was carried out on Indian coals and Part I of this standard dealing with sampling of coal was revised in 1964, in the light of the experience gained during the last eleven years.

0.3 Consequent upon the publication of IS : 436 (Part I)-1964*, an amendment (Amendment No. 3) was issued deleting all references to sampling of coal from the original standard. Also, in view of the adoption of the metric system, it became imperative that all dimensions and units in IS : 436 (Part II)-1953† be expressed in that system. This revision has been undertaken to incorporate nearest metric equivalents to the units expressed in the fps system.

0.3.1 Statistical investigation has been undertaken to examine all aspects of coke sampling and the standard will be revised in due course.

0.4 The increments and the size of the gross sample are determined by the size grade of coke. It is important that the total sample should be collected by not less than the stated number of increments which should be approximately equal in weight and evenly distributed over the consignment. It is often stated that the gross weight of the sample and the number of increments are independent of the total weight of the consignment. Nevertheless, for consignments exceeding 1 000 tonnes two or more samples are advised.

0.5 The sizes of increment and the total weight of the gross sample given in Table 1 are recommended on the basis of the size grade of coke. But

---

*Methods for sampling of coal and coke : Part I Sampling of coal (*revised*).

†Methods for sampling of coal and coke : Part II Sampling of coke.

since this is the most variable characteristic of coke, the recommended quantities of sample will be ample for the determination of other characteristics. Should any inconsistency be discovered between the general methods prescribed in this standard and the requirements of the methods prescribed with respect to a specific characteristic, the latter methods shall prevail.

0.6 In the formulation of this standard due weightage has been given to international co-ordination among the standards and practices prevailing in different countries in addition to relating it to the practices in the field in this country. This has been met by referring to the following standards:

B.S. 735 : 1944 Methods of sampling and analysis of coal and coke. British Standards Institution.

ASTM Designation D 346-35 Methods of sampling coke for analysis. American Society of Testing and Materials.

0.6.1 The Committee, responsible for the preparation of this standard, also had the opportunity of studying the draft ISO proposal for sampling of coke prepared by the Technical Committee for Solid Mineral Fuels, ISO/TC 27, of the International Organization for Standardization (ISO).

0.7 This standard is one of a series of Indian Standards for coal and coke (*see* page 13).

0.8 For the determination of particle (screen) size, the aperture size (of square holes) is based upon IS : 460-1962*. Where these sieves are not available, other equivalent standard sieves, as judged by the aperture may be used.

0.9 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960†.

1. SCOPE

1.1 This standard prescribes the methods for sampling of coke from normal consignments.

2. SAMPLING OF COKE

2.1 General — Samples collected from the surface of coke in piles, bins or wagons are, in general, unreliable, because of size segregation. Coke shall be sampled, wherever possible, in motion while it is being loaded into

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*Specification for test sieves (*revised*).

†Rules for rounding off numerical values (*revised*).

or unloaded from wagons, barges or trucks or, when it is being discharged from supply bins.

2.1.1 In case it is necessary to collect a sample of coke from the surface of loaded shipments, nine equal increments shall be taken about 30 cm below the surface. The nine sampling points shall be located as shown in Fig. 1.

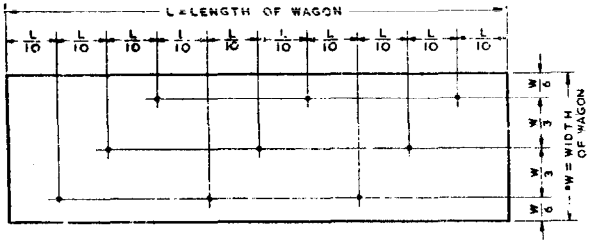


FIG. 1 LOCATION OF SAMPLING POINTS FROM SURFACE OF OPEN WAGON

2.2 Quantity Represented — A separate gross sample shall be taken for each 250 tonnes or fraction thereof or, in case of larger tonnages, for such quantities as may be agreed upon. Each lot of coke arising from a different source or known to be of different quality or size shall be sampled separately.

2.3 Weight of Increments and Gross Sample — The total weight of gross sample and the number of increments by which the sample should be collected shall be in accordance with Table 1. The minimum weight of increments shall be 1 kg (the actual increment has often to be considerably greater, as will be seen from Table 1) and the increments shall be approximately of equal weight. It is essential that the sample should be taken by not less than the stated number of increments regularly and systematically collected, so that the entire quantity of coke sampled is represented proportionately in the gross sample.

2.4 Reduction of Gross Sample and Preparation for Analysis

2.4.1 *General Precautions* — The following general precautions shall be taken:

a) The place set apart for the treatment of the gross sample shall be enclosed, roofed over, cool and free from draughts. Where this is not possible, precautions shall be taken against (1) fine wind-borne samples, (2) contamination with moisture, and (3) contamination with foreign matter.

b) The sample should preferably be reduced to 12·5 mm size by mechanical crushers or stamps, but where manual crushing is necessary, the floor, mortars, and other apparatus used should be hard, smooth, free from cracks, and of such a nature that contamination of the coke should not occur during the crushing. Appliances made of manganese steel should preferably be used.

TABLE 1 MINIMUM WEIGHT OF GROSS SAMPLE FOR COKE

(*Clause* 2.3)

SIZE GRADE OF COKE	NOMINAL SIZE	MINIMUM NUMBER OF INCREMENTS	MINIMUM WEIGHT OF GROSS SAMPLE
(1)	(2)	(3)	(4)
	mm		kg
Hard coke, large	150 to 38	50	240
Hard coke, rubble	38 „ 25	50	120
Hard coke, smithy	25 „ 12·5	50	120
Peas	12.5 „ 6·3	25	60
Coarse breeze (12·5 mm)	12·5 „ 0	25	60
Fine breeze (6·3 mm)	6·3 „ 0	25	60

c) Mechanical crushing is essential to avoid errors due to loss of moisture from large samples.

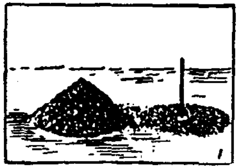
d) It is important that in the reduction of the gross sample, free ventilation of the crushed coke should be avoided.

2.4.2 *Procedure* — For reduction of gross samples, the hand method of mixing and halving by the alternate-shovel method or by coning and quartering shall be adopted as described in Appendix A.

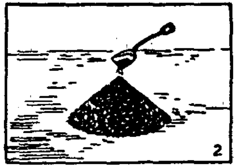
2.4.2.1 Reduce the gross sample of coke in successive stages, as illustrated in Plate 1, to 25 to 30 kg of 6·3 mm size, and then take the required number of field samples of at least 7 kg. Coke is very abrasive, and crushing by hand may best be done on a manganese steel plate using a manganese steel maul with a vertical handle. Break the 6·3 mm field sample of coke to 3·2-mm size in the laboratory, and take a sample or samples of 2 kg by coning and quartering. Reduce these in stages to pass 212-micron IS Sieve or equivalent size using a manganese steel mortar and pestle. A sample weighing 0·5 kg of the 212-micron IS Sieve material is adequate for the usual analytical tests.

NOTE — The aperture of BS Sieve 72 is within the limits laid down for the specified IS test sieve and may, therefore, be used as 212-micron IS Sieve.

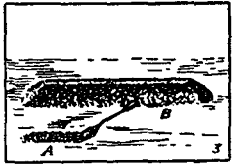
FIRST STAGE



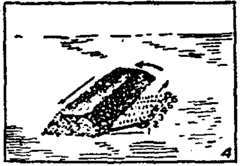
Crush sample on hard, clean surface to 25 mm size



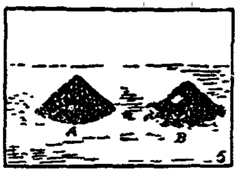
Sample crushed to 25 mm size, mixed and coned



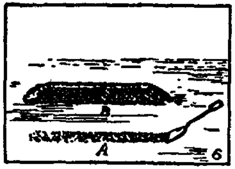
Mix by forming long pile. A — Spreading out first shovelful, B — Long pile completed



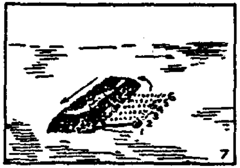
Halving by alternate shovel method. Shovelfuls 1, 3, 5, etc, reserved (Fig. 5, A) : 2, 4, 6, etc, rejected (Fig. 5, B)



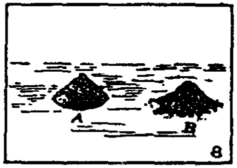
Long pile divided into two parts. A — Reserved, B — Rejected



Reserve 225 to 350 kg (Fig. 5, A). Mix by forming long pile. A — Spreading out first shovelful, B — Long pile completed



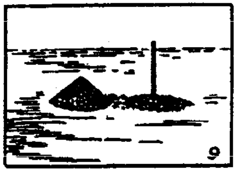
Halving by alternate shovel method. Shovelfuls 1, 3, 5, etc, reserved (Fig. 8, A); 2, 4, 6, rejected (Fig. 8, B)



Long pile divided into two parts. A — Reserve, B — Reject

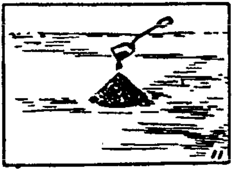
Plate 1 Illustrations of Mixing and Dividing Samples of [redacted] Coke — *Continued*

SECOND STAGE

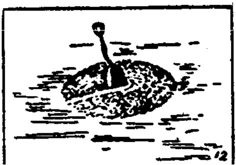


Crush 125 to 170 kg 25 mm size sample (Fig. 8, A) to 12·5 mm size

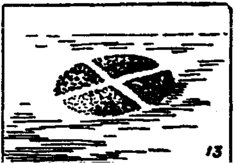
Sample crushed to 12·5 mm, mixed and coned



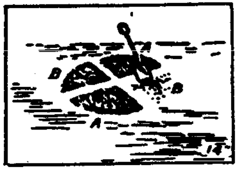
Mixed by forming new cone (repeat 3 times)



Quarter after flattening final cone

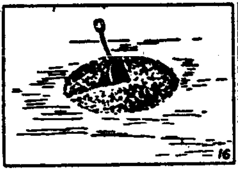


Sample divided into quarters

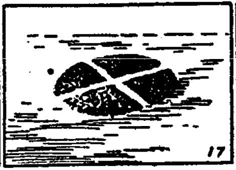


Retain opposite quarters, A, A. Reject quarters B, B

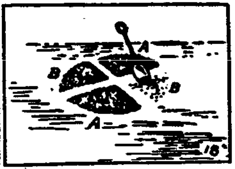
Mix A, A (Fig. 14) forming a new cone



Quarter after flattening cone



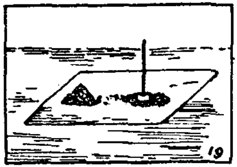
Sample divided into quarters



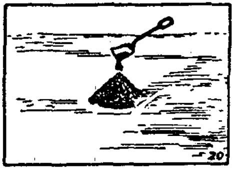
Reserve opposite quarters A, A. Reject quarters B, B

Plate 1 Illustrations of Mixing and Dividing Samples of [redacted] Coke — *Continued*

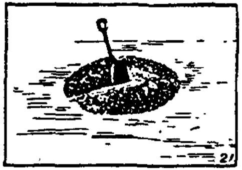
THIRD STAGE



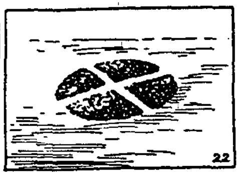
Crush 25 to 45 kg sample A, A (Fig. 18) to 6·3 mm size



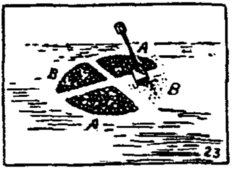
Mix by forming a new cone



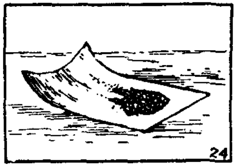
Quarter after flattening cone



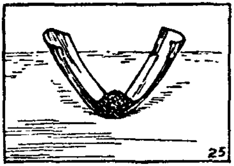
Sample divided into quarters



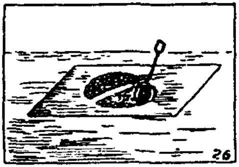
Retain opposite quarters A, A. Reject quarters B, B



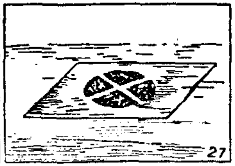
Mix A, A (Fig. 23) by rolling in tarpaulin



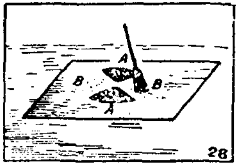
Form cone after mixing



Quarter after mixing and flattening cone



Sample divided into quarters



The laboratory sample to be taken from A, A

PLATE 1 ILLUSTRATIONS OF MIXING AND DIVIDING SAMPLES OF COKE

2.5 Ash Fusion Sample — During the procedure of crushing the gross sample in **2.4.2.1** take an additional 1-kg sample passing 12·5 mm IS Sieve at the stage indicated by Fig. 18 in Plate 1 for the determination of ash fusion. If, however, it is desired, still further, to avoid any contamination whatsoever with iron, take separate quantities of about 5 kg of the coarsely broken coke (12·5 mm and below) for the test. Do not grind the samples further in iron mills or vessels. Grind the ash obtained by incineration in an agate mortar to pass 75-micron IS Sieve.

NOTE — The aperture of BS Sieve 200 is within the limits laid down for the specified IS test sieve and may, therefore, be used as 75-micron IS Sieve.

2.6 Moisture Sample

2.6.1 For determining total moisture, draw a special moisture sample weighing approximately 25 kg, except in the case of coarse or fine breeze, in which case the moisture sample shall weigh approximately 12·5 kg. Collect the moisture sample in an air-tight waterproof receptacle with a tight-fitting lid by taking small equal parts from the increments (**2.3**) by which the gross sample is collected, the weight of increments under **2.3** being correspondingly increased to allow for this additional weight of moisture sample.

2.6.2 If possible, dry the moisture sample, without any preliminary crushing, to constant weight at a temperature of not less than 104°C nor more than 200°C. In case it is impracticable to dry the entire sample, use the following procedures:

a) *For coke appearing dry* — Crush the moisture sample rapidly to 12·5 mm size and reduce by coning and quartering to about 2·5 kg. Immediately place in a container and seal air-tight and forward to the laboratory without delay.

b) *For coke appearing wet* — Spread the moisture sample on a tared pan, weigh, and dry either in air in a warm place or on a warm or heated surface until the coke appears dry, and weigh again. Then proceed as in (a) above. Calculate the percentage of loss in air-drying and allow for it in calculating the percentage of total moisture.

$$\text{Percentage of total moisture } T = \frac{100-A}{100} \times M + A$$

where

T = percentage total moisture of the coke as received,
A = percentage of loss in air-drying, and
M = percentage moisture found in the air-dried sample sent to the laboratory.

2.7 Sampling of Coke for the Shatter Test

2.7.1 The sample shall be taken at the manufacturer's end or at the consumer's works.

2.7.2 The gross weight of the sample shall be 110 kg collected by increments distributed evenly over the whole lot of coke to be sampled.

2.7.3 Each increment shall consist of a single piece of coke, over 50 mm in size, and the pieces taken shall be representative of the size of the lumps in the consignment.

2.8 Report — Since in the report of the analysis, a brief description should be given of the method of taking the sample, by such characteristic expressions as ' belt sample ' and ' top-of-car ', information as to how the sample is taken shall be included in the description which accompanies the sample sent to the laboratory.

APPENDIX A

(*Clause* 2.4.2)

METHODS OF REDUCTION OF GROSS SAMPLES OF COKE

A-1. GENERAL

A-1.1 The mixing and reduction shall be done by hand.

A-2. HAND OPERATION

A-2.1 General Directions

A-2.1.1 Depending upon the size of the gross sample, the alternate-shovel method (**A-2.2.4**) followed by quartering shall be adopted, or only the method of quartering (**A-2.2.5**) shall be adopted. The alternate-shovel method is recommended for samples weighing about 240 kg or above. It should not be adopted for samples weighing less than 60 kg.

A-2.1.2 The two procedures are depicted in Plate 1 and detailed directions are given indicating the method of mixing, the sieve size corresponding to the weight of the gross sample, and the method of mixing and reduction to be adopted at various stages.

A-2.1.3 The weights of the samples given in Plate 1 are illustrative. A gross sample of any weight may be handled provided that the procedure corresponding to the stage related to the weight is followed and the sieve sizes indicated in Plate 1 are adopted.

A-2.2 Procedure

A-2.2.1 *Precautions* — Select a hard, clean surface, free of cracks and protected from rain, wind and direct sun. Do not let cinders, sand, chippings from the floor or any other foreign matter get into the sample. Protect the sample from loss or gain in moisture as far as possible.

A-2.2.2 *Necessary Tools and Their Use* — These include shovel, stamper, tarpaulin (3 × 3 m), hand brush and rake. Use rake for raking over coke when crushing it so that all lumps will be crushed. Sweep floor or tarpaulin clean of all discarded coke after each time the sample is halved or quartered.

A-2.2.3 *Coning* — Heap the material into the shape of a cone by pouring one shovelful of the suitably crushed material after another at the apex of the cone till the entire sample has been coned. Allow the material to slide down the sides of the cone only under the influence of gravity.

A-2.2.4 *Alternate-Shovel Reduction* — Take a shovelful from the bottom of the cone and spread it out, proceeding only in one direction, in a straight pile (3A or 6A of Plate 1) having a width equal to the width of the shovel and a length of 1·5 to 3 m. Spread the next shovelful directly over the top of the first shovelful but proceeding in the opposite direction. Repeat this piling, back and forth and occasionally flattening the pile, until the entire sample has been piled. Now, beginning at either end of the pile and shovelling from the bottom of the pile, take one shovelful and set it aside. Advancing along the side of the pile a distance equal to the width of the shovel, take a second shovelful and discard it. Advancing similarly and in the same direction, take a third shovelful and pour it on the first with a view to building up a fresh cone (**A-2.2.3**). Reject the fourth shovelful. Repeat the work round the pile, always proceeding in the same direction till the entire pile is used up and one-half of it consisting of alternate-shovelful has been coned and an equal half has been rejected.

A-2.2.5 *Quartering* — Make sure that the material is of the specified sieve size corresponding to the total weight of the sample. Crush, if necessary, and form into a cone. Flatten the cone evenly and work the apex outwards, so that it forms a low circular pile. Cut the pile into four quarters along two diameters which intersect at right angles. Retain one pair of opposite quarters and reject the other. Repeat till the size of the retained sample is reduced to the required weight.

NOTE — For samples weighing 60 kg and below, in addition to the mixing that takes place during the formation of cone, crush and mix the sample (reduced to the appropriate sieve size) by rolling the sample on a tarpaulin (3 × 3 m) or similar sheet which withstands the crushing and the rolling.

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